

TITLE

5 **REDUCTION OF FRICTION EFFECT BETWEEN POLY(ETHYLENE
 TEREPHTHALATE) PREFORMS AND BOTTLES**

CROSS REFERENCE TO RELATED APPLICATION

 This application claims the benefit of U.S.
Provisional Application No. 60/192,272 filed 27 March
10 2000.

BACKGROUND OF THE INVENTION

 The present invention relates to a unique additive
system for reducing static friction, i.e., "stiction"
in molded polyester articles and films. More
15 particularly, the present invention relates to a method
for producing low haze, transparent poly(ethylene
terephthalate) (PET) preforms, bottles, sheet and
thermoformed articles which contain such an additive
system and which exhibit improved slip performance.
20 The additive system comprises an effective amount of
(i) a fatty acid amide selected from the group
consisting of oleyl palmitamide, ethylene bis
stearamide, ethylene bis oleamide, and stearyl
erucamide; (ii) a partially or fully calcined porous
25 poly(methylsilsequioxane); and (iii) a primary and a
secondary antioxidant.

 PET homopolymer and copolymers are widely used in
rigid packaging applications, but particularly for
carbonated soft drinks (CSD) as they can form strong,
30 lightweight bottles with a satisfactory barrier to gas
transmission, primarily oxygen and CO₂. In the
production of such bottles, PET is first moulded into
small somewhat condensed shapes known as preforms,
which are relatively thick-walled test tubes with a
35 threaded neck finish, to facilitate appropriate
closure. The preforms can then be blown into the final

bottle shape (a process known as injection stretch blow
molding, or ISBM), by being heated under infrared
radiation, placed immediately into a mould, and
5 injected with a sudden blast of air. The air blast
expands the preform into the shape of the mould. One
of the problems encountered in producing preforms and
bottles from PET, however, is that these articles
exhibit a mutual sticking effect, which can make high
10 speed handling difficult on modern blowing/conveying
machines.

The effect of this sticking phenomenon with
preforms packed into a box is a lower than optimum
packing density and correspondingly higher
15 transportation cost per preform. The effect further
manifests itself when unloading these boxes onto a
blowing line, where the preforms tend not to flow
smoothly into the automatic unscramblers and other
register machinery. This can then result in less than
20 optimum preform feed rates to the blowing machine.
Bottles sticking together reduce conveying efficiency
and hence overall filling speed on a filling line, and
create further problems during bottle
25 palletising/depalletising. In sheet manufacture
sticking effects create problems in sheet unrolling and
cutting after storage, as well as with de-nesting of
thermoformed articles.

The exact origin of the observed sticking effect
has not been identified, but it may result from static
30 electricity, intermolecular attraction and/or surface
roughness.

SUMMARY OF THE INVENTION

The present invention resides in the discovery of
a unique additive system for polyester polymers which
35 improves the handling performance of articles and sheet
products produced from the polymers by reducing the

tendency of the articles and sheet products to adhere or stick. The additive system comprises an effective amount of (i) a fatty acid amide selected from the group consisting of oleyl palmitamide, ethylene bis stearamide, ethylene bis oleamide, and stearyl erucamide; (ii) a partially or fully calcined porous poly(methylsilsequioxane); and (iii) a primary and a secondary antioxidant in a ratio of 1:4, respectively.

10 According to another aspect, the present invention is a method for reducing the sticking effect, i.e., the static friction effect, or "stiction", between molded articles of PET homo- and co-polymers comprising incorporating into the polymer, at the processing stage, pre-compounded or included through a masterbatch, an effective amount of (i) a fatty acid amide selected from the group consisting of oleyl palmitamide, ethylene bis stearamide, ethylene bis oleamide, and stearyl erucamide; (ii) a partially or fully calcined porous poly(methylsilsequioxane); and (iii) a primary and a secondary antioxidant in a ratio of 1:4, respectively.

25 According to yet another aspect, the present invention is a method for producing a PET molded article, particularly bottles and other containers for carbonated soft drinks and spring water, having a reduced sticking effect which comprises the steps (1) incorporating into the polymer an effective amount of an additive system comprising a fatty acid amide selected from the group consisting of oleyl palmitamide, ethylene bis stearamide, ethylene bis oleamide, and stearyl erucamide; (ii) a partially or fully calcined porous poly(methylsilsequioxane); and (iii) a primary and a secondary antioxidant in a ratio of 1:4 at any point before molding, and then (2) molding the polymer.

The additive system imparts unexpectedly improved handling properties to transparent preforms, bottles, sheet and thermoformed articles produced from PET resin without affecting the resins's ability to consistently meet strict specifications with respect to color, taste, odor, feel and toxicity in finished bottles.

BRIEF DESCRIPTION OF THE DRAWING(S)

Fig. 1 is a graphical representation of the slip-producing effect of certain fatty acid amides in PET.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is directed to a new and useful additive system and to a method for reducing the sticking effect between PET molded articles, containers and the like, by incorporating into the PET homo- or co-polymer a unique combination of ingredients which operate to produce a desired level of "slip" while maintaining a high level of clarity and low color in use.

The term "PET" as used herein in describing the invention includes all polymeric and copolymeric forms of poly(ethyleneterephthalate). Satisfactory copolymers include those polyester copolymers which are suitable for both single and two-stage processing into various containers. The copolymer used in the examples which follow to illustrate the invention is Melinar® Laser⁺ brand PET resin (designated for convenience herein as either Laser⁺ or L⁺, and available from E. I. du Pont de Nemours and Company, Wilmington, DE), which is formulated especially for carbonated soft drink containers, and custom and mineral water containers. In order to be acceptable for carbonated soft drink containers and the like, the PET polymer must meet certain strict specifications with respect to color (i.e., low haze), taste, odor, feel and toxicity. Any

additive or additive system, therefore, designated for incorporation into the PET polymer for any purpose must not alter these strict specifications.

5 The fatty acid amides within the scope of the invention were shown to not only reduce the sticking effect of molded preforms and bottles, but also to have negligible effect on color, taste, odor, feel and toxicity in the presence of a stabilizer. Those fatty
10 acid amides are selected from the group consisting of ethylene bis stearamide, ethylene bis oleamide, oleyl palmitamide, stearyl erucamide and mixtures thereof. The fatty acid amides are incorporated into the PET in effective slip-producing amounts of from 0.01% to 20.0%
15 by weight. All of the above fatty acid amides (available from Croda Chemicals of Cowick Hall, Snaith, Goole, East Yorkshire, DN14 9AA, England) are pelletized waxy solids.

20 According to one method for carrying out the invention, the ethylene bis stearamide, ethylene bis oleamide, oleyl palmitamide, stearyl erucamide and/or mixtures thereof can be added to the PET polymer during polycondensation, i.e., after primary esterification, but before the polymer is extruded and pelletized. The
25 concentration of fatty acid amide in the polymer will be the same as the level seen in the finished PET product.

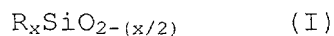
30 In yet another means for carrying out the invention, the fatty acid amide at a concentration of from 5 to 30% fatty acid amide by weight can be melt blended directly with the PET, or dry blended and then fed into a melt stream via calibrated hopper feeders, to yield a more concentrated masterbatch.

35 It is known that silica and other similar fillers, when added to PET polymers, tend to increase haze in the blown bottles, and haze is particularly detrimental

from an aesthetic standpoint. However, the invention resides in the discovery that partially or fully calcined porous poly(methylsilsequioxane) can be incorporated into the PET as part of the additive system and thereby reduce surface friction without producing objectionable levels of haze or other detrimental effects in the molded containers. Preferably, the partially or fully calcined porous poly(methylsilsequioxane) is incorporated into the PET in effective slip-promoting amounts of from 0.001% to 10% by weight.

Partially or fully calcined porous polymethylsilsequioxane for use according to the invention is prepared from Tospearl, which is commercially available from Toshiba Silicone, Japan.

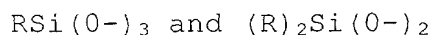
Silicone particles comprise a three-dimensional polymer chain of the formula (I):



in which x is a positive number greater than or equal to 1, preferably 1 to 1.9, more preferably 1 to 1.5, and most preferably 1 to 1.2; and R is an organic group, such as an aliphatic hydrocarbon group, e.g., methyl, ethyl, or butyl, or an aromatic hydrocarbon, e.g., phenyl, an unsaturated group, e.g., vinyl, or a mixture of two or more of these groups.

R is preferably a hydrocarbon group having 1 to 8, but preferably 1 to 5, carbon atoms, and particularly methyl. Particularly preferred silicon resin particles comprise methyl sesquioxane

Silicone particles have a cross-linked network of siloxane linkages, comprising a mixture of the following structures:



in which R is as defined above. Suitable silicone particles are commercially available from Toshiba Silicone Co., Ltd., Tokyo, Japan, under the name of "Tospearl" silicone resin particles. These particles have a three-dimensional network structure in which each silicone atom is bonded to one methyl group.

Calcining eliminates some or all of the R group, reducing the value of x. If all of the organic group is eliminated (i.e., x is 0), the particle is converted to silica (SiO_2).

To form the calcined silicone particles for use according to the invention, the particles are calcined at about 300°C to about 400°C for about 30 min to about 3 hr, preferably at about 300°C for about 45 min. Calcining may be carried out in air or in a suitable inert atmosphere, such as nitrogen. Elimination of some or all of the organic material during calcination reduces the weight of the particle. The particles typically lose from about 3% up to about 5%, but typically about 2% to about 4%, of their original weight when calcined under these conditions.

The porosity of the calcined silicone particles has been observed to enhance the adhesion of the particles to the polymer. Because the calcined silicone particles are softer (i.e., having a moh hardness of 3) than glass particles (i.e., moh hardness of 6), they are less prone to scratch the bottles during transport.

An analysis of calcined Tospearl particles was carried out as described below.

Tospearl Analysis

Volume distributed median particle diameter, and particle size distribution ratios D_{25}/D_{75} and D_{10}/D_{90} were measured using Microtrac.

BET specific surface area was measured by multi-point nitrogen adsorption using a Micromeritics ASAP 2400. Relative pressures between 0.05 and 0.21 were
5 used, and the outgassing conditions were 1 hour at 140°C with nitrogen purge (1 to 2 litres/ hour).

Skeletal density was measured by helium pycnonmetry using a Micromeritics Accupyc 1330.

The ratio of methyl groups to silicon atoms was
10 measured by ^{29}Si magic angle spinning NMR spectroscopy. The spectrum was acquired on a Bruker MSL200 NMR spectrometer operating at a frequency of 39.73 MHz for ^{29}Si . The magic angle was set using KBr and the
15 spinning speed was 5050 Hz. The NMR free induction decay consisting of 2K complex data points was acquired using the single pulse excitation pulse sequence together with high power ^1H decoupling where the ^1H
20 decoupling field was of the order of 70 kHz. The spectral width was 20 kHz, ^{29}Si pulse length 5.5 μs (90°) and recycle delay 60 s. 1000 transients were accumulated. Data processing was carried out with the Bruker LB parameter equal to 60 Hz, followed by Fourier transformation, phasing, baseline correction and
integration using the Bruker software EP-I routine.

25 Calcining

Silicone resin particles (Tospearl, supplied by Toshiba Silicone Co Ltd., Japan) were calcined by heating in an oven at approximately 300°C for approximately 4 hours in an atmosphere of air in order
30 to produce filler particles for use in the present invention. The resultant calcined silicone resin particles exhibited the following characteristics :

- (i) volume distributed median particle diameter
= 2 - 3 micron

- (ii) particle size distribution ration $D_{25}/D_{75} =$
0.6 - 0.9 micron
- (iii) particle size distribution ration $D_{10}/D_{90} =$
1.1 micron
- (iv) BET specific surface area = 48 - 60 micron
 m^2/g
- (v) skeletal density = $2.06 g/cm^3$
- (vi) ratio of methyl groups to silicon atoms =
0.2

The presence of fatty acid amides in PET can present problems during processing at elevated temperatures due to thermal-oxidative degradation. This can be overcome according to the invention through the use of selected primary and secondary antioxidants as stabilizers. Primary antioxidants are selected to interrupt the propagation of radicals associated with thermal-oxidative degradation and of the general types of hindered phenols and aryl amines. Hindered phenols and multifunctional hindered phenols are preferred primary stabilizers.

Secondary antioxidants are intended to stop degradation reaction initiation by removal of hydroperoxides, and they are generally selected from phosphorous or sulphur-containing compounds. Phosphites or phosphite esters, and particularly aryl phosphite esters, are preferred for use according to the invention.

Primary and secondary antioxidants employed in combination according to the invention are more effective than when either is used independently. The use of these materials is established here for the particular system incorporating the "stiction" additives claimed. The specific combination described herein (1 part hindered phenol:4 parts phosphite ester)

was found to provide superior color compensation. The combination is incorporated during preparation of the masterbatch. Then, all of the additives can
5 conveniently be fed simultaneously by feeding the masterbatch via calibrated feeders into the PET melt stream. The concentration of primary antioxidant can be from 0.14 to 28% by weight, and the concentration of secondary antioxidant can range from 0.04 to 8.0% by
10 weight.

In a preferred embodiment the primary stabiliser, i.e., antioxidant, is tetrakis[methylene(3,5-ditert.butyl-4-hydroxy-hydrocinnamate)] methane, which is available commercially as Irganox 1010 (Ciba, Basel,
15 Switzerland), and the secondary stabilizer is (2,4-di-tert.butylphenyl)phosphite, which is available commercially as Irgaphos 168 (Ciba Speciality Chemical, Ltd., Basel).

Examples

20 Measurements were conducted to identify candidates for use as anti-stiction additives in bottle preforms and to determine at what concentration an additive would produce an optimum anti-friction, or slip-producing, effect.

25 A preform stiction apparatus was designed to provide a reproducible measurement of the adhesive force between PET preforms. This can identify differences between preforms of different compositions. In Laser⁺ preforms a stearyl erucamide bead additive
30 was found to provide the minimum level of adhesion.

Test Procedure

A range of Laser⁺ PET formulations were injection moulded into parison tubes, or preforms. The moulding was carried out at 260°C under identical injection
35 moulding conditions. The formulations comprised

representative samples with different additives
selected from oleyl palmitamide and stearyl erucamide
at a concentration of 0.1% by wt and one control
5 formulation (Laser⁺ with no additive). The preform
samples were moulded using a Negri Bossi NB90 molding
machine. In addition, an extra batch of Laser⁺ control
preforms were moulded using a Husky XL 160 molding
machine for comparison.

10 Each preform sample was allocated an
identification number. The start and finish time/date
of moulding was recorded allowing the time of moulding
of each preform to be estimated. The preforms were
wrapped in aluminium foil and handled as little as
15 possible at all stages. In order to control the
environmental effects and to avoid surface
contamination, the injection moulded preforms were
stored in temperature and humidity controlled
laboratory conditions within 15 minutes, and the
20 measurements were recorded in the controlled
environment.

The model of adhesive friction used for the
evaluation was as follows:

*Adhesive shear force = interfacial strength per unit
25 area X real area of contact*

Interfacial shear strength per unit area is a good
indication of the strength of the adhesion, and
therefore the stiction, in a surface-to-surface
contact. The adhesive shear force makes up part of the
30 measured friction force, and deformation also makes a
contribution. The real area of contact was generally
approximated by using:

real area of contact = normal force/hardness.

According to this approximation, the adhesive
35 coefficient of friction will be independent of both

contact geometry and normal force. However, in the case of PET bottles, the combination of high attractive forces across the contact, low hardness and easy
5 deformation of the wall may lead to greater true areas of contact those predicted and therefore greater coefficients of friction. So to get results that reflect the value of the interfacial strength the contact was kept consistent from test to test and
10 sample to sample.

A crossed cylinder apparatus gave a good reproducible contact geometry (similar to sphere on flat). The preforms had a relatively small diameter and were quite stiff, deforming only slightly with the
15 normal load, giving a small contact spot and shear forces of a magnitude that were easily measurable.

Two cylindrical preforms were mounted at 90° to one another and pressed together with a known force using a solenoid. This gave a circular contact point
20 similar to that observed in a sphere against flat arrangement. The contact area will be small as the preform walls are rigid and the preform diameter is small. The configuration allows the parallel walled section of the preforms to be brought into direct
25 contact with one another. This geometry is representative of the conditions under which stiction is observed in practice. An Instron test frame then controls the movement of one preform across the other at a fixed load and rate, while friction force, normal
30 force and position can be simultaneously recorded.

Sample preforms were loaded together with a force of 10 N. This load was selected to optimize the accuracy of the technique. A 10 N force can be applied easily and with accuracy without the need for a
35 sensitive, delicate and/or expensive load cell to monitor the normal force. This normal force gives a

magnitude of friction force which can be read accurately with standard 100 N Instron load cells. A higher force has been adopted in these tests than in normal applications because the adhesive force is related to the normal force only through its influence on the contact area (especially in the case of static friction, where frictional heating is not a factor). Thus, the relatively high magnitude of the normal force makes testing easier and more accurate without rendering the data inapplicable to the application. The normal force was applied for one minute before starting each experiment. Contacts are known to change with time, particularly in polymers where viscoelastic effects can be significant, and so a constant loading time was a prerequisite. The sliding speed employed was 200 mm/min, and the traverse length was 20 mm (all within the parallel wall range of the preform samples). Experiments were carried out over five to ten days following moulding, and repeat tests were carried out at each time point, using a minimum of three pairs of preforms tested for statistical significance. Experiments were carried out over five to ten days following moulding also because stiction may well be influenced by time dependent phenomena, such as, for example, migration of low molecular weight material or additives to the surface.

Static coefficient friction is regarded as being a direct measurement of stiction since it is a measure of the adhesion between the preforms. Stiction can be said to have occurred when the static coefficient of friction has a greater value than the dynamic coefficient of friction. The results are shown graphically in Fig. 1, from which it can be seen that preforms which included a fatty acid amide exhibited improved slip properties over the control, and stearyl erucamide exhibited the best slip performance.

Additional data obtained from subsequent trials is shown below in Table 1.

Table 1

Sample ID	Test A Results		Test B Results for PET Film	
	Static Average COF	Dynamic Average COF	Static Average COF	Dynamic Average COF
L+ Control	1.314	0.877	3.646	0.9116
L+ 0.1% ER*	0.564	0.416	1.315	0.407
L+ 0.2% ER*	0.602	0.458	0.2416	0.473

5 *ER = stearyl erucamide

Additional tests were conducted on unoriented PET cast sheet using an Instron slip instrument to determine slip enhancement obtained from a combination of stearyl erucamide and Tospearl. COF values can be
10 seen in Table 2.

Table 2

	Static Friction	Dynamic-Friction
Laser* (control)	5.14	1.75
Laser*+0.1 % ER* + 30 ppm Tospearl	0.50	0.40
Laser* + 0.2 % ER* + 8 ppm Tospearl	0.65	0.40
Laser* + 8 ppm Tospearl	0.65	0.43

* ER = stearyl erucamide

The fatty acid amide and Tospearl can be added to the polymer either during polycondensation or during
15 direct esterification. However, fatty acid amides, as discussed above, are not thermally stable at typical PET processing or drying conditions. The presence of a fatty acid amide, such as stearyl erucamide, in PET can produce a slightly more green (increase in minus
20 b*), and yellow (more positive b*) color in the polymer and in molded articles produced from the polymer. Thus, the invention includes selection of a compatible thermal stabilizer to complete the additive system. As described above, a primary and a secondary antioxidant

are employed in combination as a stabilizer package according to the invention, and this results in more effective color control than when either is used independently. The specific and preferred combination is 1 part hindered phenol:4 parts phosphite ester. The combination is incorporated during preparation of the masterbatch. Then, all of the additives can conveniently be fed simultaneously by feeding the masterbatch via calibrated feeders into the PET melt stream. The concentration of primary antioxidant can be from 0.14 to 28% by weight, and the concentration of secondary antioxidant can range from 0.04 to 8.0% by weight.

15 Color

Tristimulus colour measurement was carried out by using the colorgard system and tristimulus colorimeter whereby colour is expressed as L*, a* and b*. L* is the lightness variable, and a* and b* are the rectangular colour co-ordinates. Positive values of a* signify redness, negative values greenness, and positive values of b* signify yellowness, negative values blueness.

Polymer test samples, i.e., batches, were prepared which contained 0.2% by wt stearyl erucamide (ER) and 30 ppm Tospearl along with concentrations of the stabilizers shown in Table 3.

Table 3 - Sample Identification

Sample Number	Wt% Irgafos 168	Wt% Irganox 1010
1	0.28	0.08
2	0.08	0.28
3	0.16	0.16
4	0.14	0.04
5	0.21	0.06
6	0.245	0.07
7	Nil	Nil
Laser Control Batch		
8	Nil	Nil
0.2%ER ¹ & Tospearl		

Plaques were prepared from each of the sample polymers, and color measurements were obtained. The results are shown in Table 4. Plaque colour measurements which highlight the effect of fatty acid amides are shown in Table 5.

Table 4

4mm Plaque Colour from polymer chip samples dried @ 175°C for 5 hours

	L*	a*	b*	YIE	Haze
Sample 1	73.90	-0.30	5.82	10.86	16.2
Sample 2	73.22	-0.66	7.40	13.76	23.2
Sample 3	74.87	-0.60	6.83	12.51	17.9
Sample 4	75.23	-0.22	6.23	11.41	16.3
Sample 5	74.48	-0.40	5.91	10.94	16.2
Sample 6	74.46	-0.46	6.74	12.39	18.2
Sample 7	73.63	0.24	3.24	6.14	11.4
Sample 8	71.52	-0.51	9.91	18.44	18.8

Table 5.

Plaque Colour Measurements Highlighting The Effect of Fatty Acid Amides.

	L*	a*	b*	YIE	Haze	Si Content
Virgin L* - Not compounded through extruder	77.60	-0.39	3.56	7.78	10.5	0
L* Control - Compounded through extruder	78.99	-0.27	4.03	8.81	11.2	0
No ER ¹ + 168 ² + 1010 ³ + Tospearl	78.64	-0.33	4.09	8.92	17.6	11ppm
0.2% ER ¹ + 168 ² + 1010 ³ + 40ppm Tospearl	80.62	-0.86	6.3	12.94	12.9	10ppm
Oleyl Palmitamide + 168 ² + 1010 ³ + Tospearl	79.51	-0.78	5.81	12.11	11.8	8ppm

5 ¹ ER = stearyl erucamide @ 0.2wt%

² Irgafos 168 @ 0.28wt%

³ Irganox 1010 @ 0.08wt%

Additional color measurements are shown in Table 6 wherein 0.28wt% 168², 0.08wt% 1010³ and 30ppm Tospearl was added to each polymer sample unless stated otherwise.

Table 6.

Plaque Colour Measurements Highlighting The Effect of Fatty Acid Amide Loading

	L*	a*	b*	YIE	Haze	Si Content
Virgin L* - Not compounded through extruder	77.60	-0.39	3.56	7.78	10.5	0
L* Control - Compounded through extruder	78.99	-0.27	4.03	8.81	11.2	0
No ER ¹ + 168 ² + 1010 ³ + Tospearl	78.64	-0.33	4.09	8.92	17.6	11ppm
0.1% ER ¹ + 168 ² + 1010 ³ + Tospearl	78.83	-0.76	5.32	11.16	16.7	10ppm
0.2% ER ¹ + 168 ² + 1010 ³ + Tospearl	80.62	-0.86	6.3	12.94	12.9	10ppm
0.5% ER ¹ + 168 ² + 1010 ³ + Tospearl	78.23	-1.98	9.87	19.66	21.7	16ppm

15

Additional color measurements are shown in Table 7 wherein 0.28wt% 168¹, 0.08wt% 1010² and 30ppm Tospearl was added to each sample unless stated otherwise.

Table 7

Plaque Color Measurements Highlighting The Effect of
Tospearl.

	L*	a*	b*	YIE	Haze	Si Content
Virgin L* - Not compounded through extruder	77.60	-0.39	3.56	7.78	10.5	0
L* Control - Compounded through extruder	78.99	-0.27	4.03	8.81	11.2	0
No Tospearl + 0.2% ER ¹ + 168 ² + 1010 ³	78.88	-1.07	6.19	12.73	9.18	0
Tospearl + 168 ² + 1010 ³ + No ER ¹	78.64	-0.33	4.09	8.92	17.6	11ppm
40ppm Tospearl + 0.2% ER ¹ + 168 ² + 1010 ²	80.62	-0.86	6.3	12.94	12.9	10ppm
80ppm Tospearl + 0.2% ER ¹ + 168 ² + 1010 ³	79.63	-0.8	5.5	11.42	16.3	14ppm
130 Bead Tospearl + 0.2% ER ¹ + 168 ² + 1010 ³	79.19	-1.06	6.96	14.33	18.1	9ppm
145 Bead Tospearl + 0.2% ER ¹ + 168 ² + 1010 ³	79.13	-1.05	6.43	13.24	11.4	7ppm

- 5 Additional color measurements are shown in Table 8
wherein 0.28wt% 168², 0.08wt% 1010³ and 30ppm Tospearl
was added to each sample unless stated otherwise.

Table 8

- 10 Colour comparison of thin film, thick sheet,
bottles and thermoformed cups containing fatty acid
amide/Tospearl variants versus control samples.

Type	Sample	L*	a*	b*	YIE	Haze
Thin films	L* Control	88.97	0.06	4.21	8.63	0.99
	L* + 50ppm Tospearl	89.40	0.06	4.19	8.55	1.90
	L* + 210ppm Tospearl	88.98	0.06	4.19	8.57	2.26
Thick film	L* + 30ppm Tospearl	89.39	0.08	4.22	8.62	1.97
Thin Sheets	L* Control	89.34	0.04	4.19	8.53	0.63
	L* + 50ppm Tospearl	89.24	0.06	4.19	8.56	3.03
Thick Sheet	L* + 30ppm Tospearl	88.38	-0.04	4.56	9.29	2.31
1mm Plaques	L* + 0.1% ER ¹ + 30ppm Tospearl + Stabilisers	85.90	-1.02	7.62	14.75	9.79
	L* + 0.2% ER ¹ + 30ppm Tospearl + Stabilisers	85.71	-0.94	7.59	14.80	10.4
2L Bottles	L*	87.57	-0.03	4.69	9.62	5.85
	L* + 8ppm Tospearl	87.84	0.00	4.57	9.39	7.76
	L* + 0.1% ER ¹ + 30ppm Tospearl	87.58	-0.03	4.62	9.47	5.75
Thermoform Cups	L* Control	89.15	0.02	4.71	9.55	2.09
	L* + Stiction additive package	89.07	0.09	4.40	9.02	2.47

Table 9

Effect With and Without 168² on Plaque Color.5 Degradation of plaques over time

L⁺ used with and without 0.28wt% Irgafos 168 but including 0.2wt% ER¹, 0.08wt% Irganox 1010 and 30ppm 120 bead Tospearl. The absence of Irgafos 168 in the polymer is detrimental to color. The absence of

10 Irgaphos 168 results in the presence of hydroperoxides which facilitate degradation

	L*		a*		b*		YIE*		Haze	
	+168	-168	+168	-168	+168	-168	+168	-168	+168	-168
2 hrs drying	78.40	79.54	-1.45	-2.03	5.79	8.24	11.60	16.06	18.7	20.2
3 hrs drying	78.44	80.00	-1.19	-1.71	5.65	6.81	11.52	13.31	18.6	16.9
4 hrs drying	79.95	79.09	-1.40	-1.53	6.67	6.49	13.31	12.92	18.7	17.7
5 hrs drying	78.52	79.33	-1.43	-1.52	6.11	6.65	12.29	13.24	18.6	17.6